STEM and HRTEM techniques for investigation of cobalt recovery temperature dependence under ion beam irradiation

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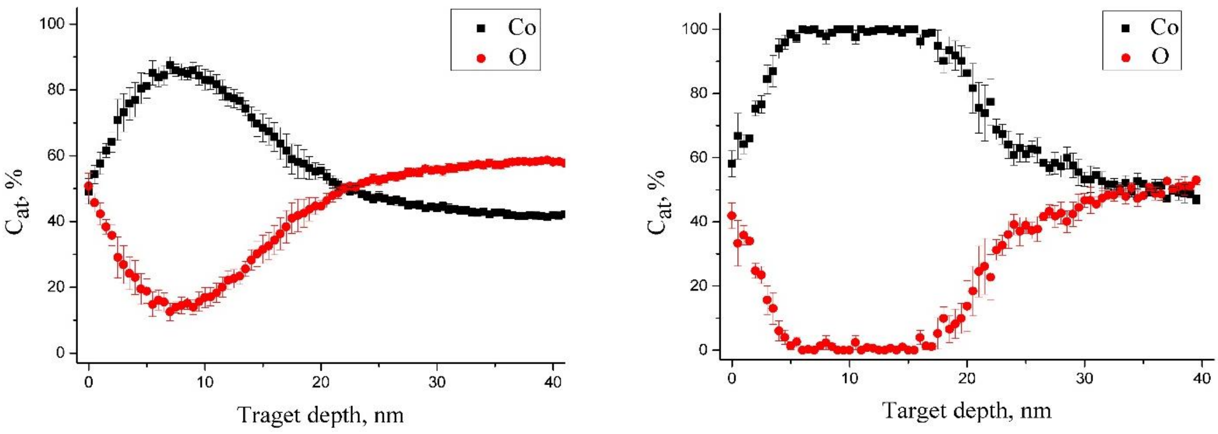
Currently, there is a great interest in the creation of nanostructures based on ferromagnetic and antiferromagnetic metals. First of all, such interest is associated with the possibility of using it in magnetoelectronics to generate magnetic memory elements. It is known that for production a magnetic medium with a high recording density, it is necessary to form a special medium consisting of individual magnetic granules (single-domain bits) with identical size from 15 to 30 nanometers, shape and orientation (pattern magnetic media) [1]. Moreover, in such structures is important to provide magnetization stability of the individual bits when the external magnetic field is turned off. Creating an antiferromagnetic layer can solve the problem of individual bits stability, for example, CoO - layer along the ferromagnetic Co-bit boundary.

Radiation technology of creating functional nanoelements has been actively developing in the NRC "Kurchatov Institute" during the last ten years. However, it is important to note, that the atomic composition and physical properties controlled modification of selected regions of thin-film materials (ferromagnets, insulators, conductors, superconductors) occurs due to low-energy ions irradiation with various composition (protons, oxygen, nitrogen ions, etc.) at different doses. The problem of determining the irradiation conditions (energy of incident ions, irradiation dose and substrate temperature during irradiation) for functional domains formation with specified composition and properties becomes actual.

In this study, we offer the radiation technology of selective removal of oxygen atoms (SRA) [2] from cobalt oxide Co_3O_4 to create a high-density pattern media. Selection of irradiation conditions and predicting physical properties of modified materials requires structure and chemical composition data after irradiation. Modern analytical transmission electron microscopy makes it possible to obtain quantitative information about chemical composition of samples with a local sensibility up to 1 nm. Analysis of Co_3O_4 films modification degree in depth was carried out using electron energy loss spectroscopy (EELS) in convergent beam mode (STEM mode). Phase analysis of films irradiated at different doses was carried out using high-resolution bright-field images on transmission electron microscope Titan 80 - 300 operated at 200 kV. Preparation of films cross-section was performed at FIB Helios Nanolab 650 facility.

The SRA process was realized by irradiating the initial cobalt oxide films with 1 keV protons at temperature of 20°C and 100°C at doses: $0.94\cdot10^{18}$, $1.87\cdot10^{18}$ и $3.75\cdot10^{18}$ ions/cm^2. An estimate of the chemical composition changes in film depth was carried out after Co_3O_4 thin films proton irradiation at different fluences and substrate temperatures. Results of substrate temperature influence during irradiation are shown in Figure 1. Figure 1 demonstrates elements distribution profiles in depth of irradiated films to a dose of $3.75\cdot10^{18}$ ions/cm^2 at substrate temperatures of 20°C and 100°C. As can be seen, the Co_3O_4 film irradiated at 100°C substrate temperature at given dose is restored to pure cobalt at a depth of 17 nm, as compared with irradiation at a temperature of 20°C. This is due to the faster release of knocked out oxygen atoms from the sample during the process of selective removal oxygen atoms from Co_3O_4 under proton irradiation. Furthermore, it was found that at both irradiation temperatures elements depth distribution profiles are non-monotonically, which is due to the nonmonotonic distribution of the damaging dose during irradiation. The direct correspondence of cobalt recovery profile to the dose distribution profile in target depth confirms the radiation nature of selective removal process of atoms.



(a) (b)

Figure 1. Elements depth distribution profiles of the Co_3O_4 film irradiated with 1 keV protons (calculated from EELS data) for dose $3.75\cdot1018$ ions/cm^2 at different temperatures: (a) 200°C; (b) 1000°C.

Phase identification analysis performed by Fourier - transform of corresponding grains in HRTEM image completely agrees with EELS data. Figure 2 showed that the grain of Co_3O_4 film irradiated at a dose of $3.75\cdot10^{18}$ ions/cm^2 at 100^0 C substrate temperature corresponds to the phase of pure Co hexagonal system (P63/mmc) with a lattice parameter a=b=0.2514 nm, c=0.4105 nm.

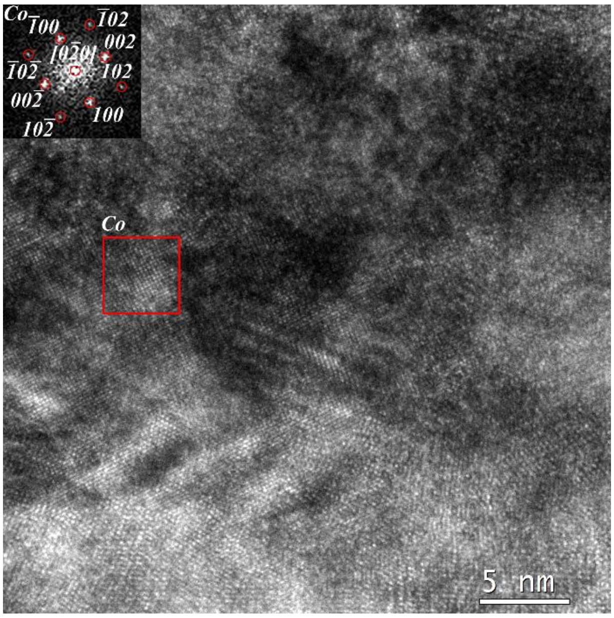


Figure 2. Bright-field HRTEM image of Co_3O_4 film cross section irradiated with 1 keV protons with dose $3.75\cdot1018$ ions/cm^2 at 1000°C, diffraction (inset).

1. E.Z. Meilikhov, R.M. Farzetdinova, *FTT* **56**, 2326 (2014).
2. B.A. Gurovich, E.A. Kuleshova, K.E Prikhodko, et al., *Journal of Magnetism and Magnetic Materials* **322**, 3060 (2010).